UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
230 SOUTH DEARBORN ST.
CHICAGO, ILLINOIS 60604

REPLY TO THE ATTENTION OF:

5SMQA

MEMORANDUM

DATE: FEB 08 1990

SUBJECT: Review of the First Revision, PRP-Lead Quality Assurance Project Plan for Phase II Remedial Investigation/Feasibility Study Activity at the American Chemical Services Site in Griffith, Indiana

FROM: James H. Adams, Jr., Chief
Quality Assurance Section

TO: James Mayka, Chief
Illinois/Indiana Section

ATTENTION: Robert Swale, RPM

We have reviewed the first revision, PRP-Lead Quality Assurance Project Plan (QAPjP) for Phase II Remedial Investigation/Feasibility Study (RI/FS) activities at the American Chemical Services (ACS) site in Griffith, Indiana, Which was received by the Quality Assurance Section (QAS) on January 2, 1990 (QAS Log-In No. 1120). This subject QAPjP is not approvable because most of the required Standard Operating Procedures (SOPs) are not acceptable. This subject QAPjP will not be approved until deficiencies listed in this memorandum are properly addressed.

Our comments on the current QAPjP are summarized as follows:

I. SOP for Low Detection Limits - Volatile Organics

A. The first 12 pages, which cover the internal laboratory operations such as creating file name, etc., shall not be part of the SOP, and shall be deleted.

B. The following deficiencies shall also be corrected:

1. The concentration of stock standard solutions shall be specified. It is not acceptable to identify the specific standard solution in terms of the laboratory code number (i.e., Standard #349). If it is necessary to use the laboratory code for the convenience of daily laboratory operation, we suggest that the actual concentration of that solution be identified in a parenthesis - for example,

solution #4000 (200 ug/L).

2. It is stated under "Standard Preparation" in page 3 of 10, that two 10 ml syringes will be used to deliver 20 ml of standards and samples into the purging device. This is not acceptable. We require that a 20- or 25 ml syringe shall be used.

3. Under "SPCC Criteria" in page 5 of 10, it is stated that the SPCC criteria for bomoform and 1,1,2,2-tetrachloroethane are waived for this analysis. This is not acceptable. The bomoform and 1,1,2,2-tetrachloroethane shall not be waived. The Relative Response Factor (RRF) for these two compounds shall be at least 0.150. Please make the same correction in page 8 of 10.

4. In page 5 of 10, the criteria for the continuing calibration check shall be revised as follows:

   a. The Percent Relative Difference (%RPD) for any compounds shall not be greater than 25% of the initial calibration.

   b. The standard solution used for continuing calibration check shall include all compounds of interest at concentration of 20 ug/L.

   c. The continuing calibration check shall be done daily at the beginning of the day before analysis of any samples, and at the beginning of each 12-hour shift.

5. In page 8 of 10, under "Sample Preparation", the 10-ml syringe shall be replaced with 20- or 25-ml of syringe.

6. A separate section shall be added to address the criteria to be used for the qualitative identification of compounds.

7. The frequency of analyzing method blank and continuing calibration check standards shall be specified.

8. Attachment 1 shall be revised to include the actual quantitation limits the responsible laboratory can achieve.

9. The level of matrix spike and surrogate spike shall be done at concentrations of 20 ug/L.

C. Use the attached SOP example as reference to revise this SOP.

II. <u>SOP for Low Detection Limits - Extractables</u>

A. Please identify the actual concentrations of each spike and surrogate

standard solutions. See comment I-B-1 of this memo.

B. The concentration of the surrogate spike and matrix spike shall be done at 20 ug/L for base/neutral compounds and 40 ug/L for acids. Please address them.

C. A table listing the target compounds along with the required detection limits shall be included in the SOP.

D. The required quality control, which includes the analysis of method blank, matrix spike/matrix spike duplicate, continuing calibration check, and their frequencies shall be properly addressed.

E. A separate section shall be added to address the criteria to be used for the qualitative identification of compounds.

F. See Comment I-C of this memo.

III. SOP for Low Detection Limits - Pesticides/PCBs

A. Please identify the actual concentrations of each spike and surrogate standard solutions. See comment I-B-1 of this memo.

B. The concentration of the surrogate spike shall be done at 0.2 ug/L. Please address them.

C. The level of matrix spike shall be done as follows:

Compound	Concentration (ug/L)
Lindane	0.04
Heptachloro	0.04
Aldrin	0.04
Dieldrin	0.10
Endrin	0.10
4,4'-DDT	0.10

D. The required quality control, which includes the analysis of method blank, matrix spike/matrix spike duplicate, calibration check,and their frequencies shall be properly addressed.

E. A analysis sequency including the steps of calibrations, calibration checks, shall be addressed.

F. Please provide the procedure to be used to quantify the PCBs.

G. A table listing the target compounds along with the required detection limits shall be included in the SOP.

H. A separate section shall be added to address the criteria to be used for the qualitative identification of compounds.

I. See comment I-C of this memo.

IV. SOP for Alkalinity

A. The procedure, including the equation, to be used for calculating the analytical results shall be properly addressed.

V. SOP for Total Organic Carbon in Soils

A. It is indicated that the instrument has three ranges of sensitivity; however, it is not clear whether all three ranges are interchangeable. Please clarify it. If they are not interchangeable, how the calibration to be done when the range of sensitivity is changed shall be documented in the SOP.

B. For the analysis of soil samples, what is the standard to be used for calibration and continuing calibration check ? Please identify the standard to be used, including the amount to be used in the SOP.

VI. SOP for Chloride Analysis

A. The matrix spike level specified in the SOP is not acceptable because the spike level shall determined based on the concentration of chloride detected in the sample. Please address it properly by specifying the spike level for both samples with low/no chloride detected, and sample with high concentration of chloride.

VII. SOP for Total Cyanide Analysis

A. Please identify the preparation and the concentration of the LCS standard solution.

VIII. SOP for Mercury Analysis

A. The equation used for calculating the %recovery appears to be incorrect. Please correct it accordingly.

IX. SOP for Total Kjeldahl Nitrogen (TKN)

A. This SOP is not applicable to this project, and shall be deleted.

X. Table 3 of the QAPjP

A. Please revise this table to include 1 matrix spike/matrix spike duplicate (MS/MS) for sediment sample designated for the analysis of pesticides/PCBs.

To expedit the QAPjP approval process, we strongly suggest that RPM shall forward QAS' review memo to contractors in a timely fashion (i.e., 2 days after receiving the memo). We estimate that 7 working days shall be adequate to address all of the deficiencies mentionmed above.

We also strongly suggest that, after PRP's QAPjP preparer has reviewed the QAS comments, a QAPjP meeting or conference call shall be held between QAS, RPM, QAPjP preparer, and other concerned parties, including laboratory personnel. The QAPjP meeting or conference call will improve communication between QAS and all concerned parties, and will thus minimize the number of comments on, or revision of QAPjP. As a results of the conference call/meeting, the QAPjP approval process can be shortened. Furthermore, we would like to receive a copy of the RPM's memo to QAPjP preparer if there is any deviation from QAS' original comments.

REVISED SOP FOR AUTOANALYZER

AUTOANALYZER

Scope and Application: Ions can be readily analyzed by a flow-injection autoanalyzer. The flow injection design gives the system excellent washout characteristics, to prevent carry over and cross contamination. The autoanalyzer is generally more sensitive and accurate than the manual wet-chemistry techniques.

Method: Flow injection

References: Lachat Instruments, 1986.

Sample Handling: See separate SOP's for requirements.

Reagents and Apparatus:

1. Lachat 3-channel autoanalyzer
2. Stock and standard ion solutions
3. Class A volumetric flasks
4. Class A volumetric pipets
5. Milli-Q water
6. Required interference filters
7. Disposable 4 mL cups
8. Automatic sampler
9. Proportioning pump
10. Injection module
11. Colorimeters
12. Manifolds
13. Columns - if needed
14. Helium gas
15. Computer
16. Printer

Procedure:

A. Instrument Set-Up

1. Depress red power switch on power strip located behind the computer terminal. This will turn on the computer, the screen, and the printer.

2. Depress red power switch on rear power strip on Lachat system.

3. Select manifold and make appropriate hydraulic connections.

   Hydraulic connections:

a. Use correct sample loop length to connect. Lines 1, 4.

b. Line 2 is carrier line.

c. Line 3 goes to manifold.

d. Line 5 goes to waste container.

e. Line 6 comes from sample probe.

f. Connect manifold to flow through cell.

Tension levers should be up when pump tubing is inserted. Snap pump tubing cartridges into place.

4. Insert correct filter.

5. Pump Milli-Q water through lines for 5 minutes by depressing the pump ON button. Check for leaks.

6. Computer - At the C> type in "quikcalc". This calls up the Lachat software and puts you at the master menu. Press <enter>.

7. Put lines into reagents and/or degassed Milli-Q water.

8. Computer - Select "Load/Stop Background Method" on the master menu. Press <enter>.

9. Select appropriate method. Press <enter>.

10. Printer should be set at FONT 0.

11. Pump reagents until a steady baseline is achieved.

12. When using a method with a column (SO_4 or NO_3), the column may be inserted at this point. See method SOP's for more details.

13. For each analytical channel, adjust zero knob so that the baseline is near the bottom of the screen (between .000 - .030).

14. Adjust gain while injecting top standard.

    a. Place autosampler probe into the highest standard bottle.

    b. After 20-30 seconds, press cycle button on front panel so that LED light is red. This is the load position.

    c. After 25 seconds (or less depending on sample loop size), press cycle button so that LED light is green. This is the inject position.

d. Adjust gain knob on detector so that peak reading on the colorimetric is 1.700-1.950.

e. Repeat until gain is properly adjusted.

f. Wipe probe and replace the autosampler probe into the sampler.

15. Select menu item by going into foreground. (Press and hold Alt key, then press Esc key).

    a. Select "Sample Tray Information and Start Analysis" on master menu. Press <enter>.

    b. Press <enter> or type in sample tray reference number if it is a tray which has already been typed in.

    c. Enter tray ID and operator. Check "Display Standards Position in Tray" to insure the tray is set-up properly.

    d. Select "Enter Sample ID's". Press <enter>.

    e. Type in sample information. Check standards will automatically be placed in the tray information portion.

    f. Press Esc once to return to menu.

16. Put tray with samples in appropriate cup locations on autosampler. Position try to the cup containing standard A (usually #35 or so). Select "Start Analysis." Press <enter>.

17. The second screen will ask if the tray has standards or not. If you standardized the first tray of the run and all the check standards are within QC ranges, recalibration for the next tray is <u>not</u> necessary. Select appropriate option. Press <enter>.

18. Press Alt, Esc keys together, to get back to background to view the calibration peaks.

    After calibration is complete:

    - go into the foreground (Press Alt, Esc keys)
    - select "display calibration graph" (Press <enter>)
    - review the data
    - return to the background (Press Alt, Esc keys)

- press "G" for good calibration. Analysis will continue.

- press "R" for re-calibration. Remember to refill standard cups and reposition sample tray before pressing "R"!

B. Instrument Shut-Down

1. Press Alt/Esc keys to get to the foreground. Select "Load/Stop Background Method". Press <enter>. To question-"Stop background (Y/N)?" Press "Yes". Press Esc key to return to main menu.

2. If column is used, stop the pump and disconnect from manifold.

3. Pull lines from reagents into a wash beaker of D.I..

4. Pump D.I. through lines for 2-5 minutes.

5. Pump air through lines until manifold is dry.

6. Turn off pump.

7. Release tubing cartridges and lower tension levers. Release tubing.

8. Turn off main switch on rear power strip.

9. Empty and rinse waste containers, if necessary.

10. Perform back-up on current data files, once a week. (See Section C)

11. Turn off the computer and printer.

C. Backing-up the Data Files

1. Exit to DOS

2. At C> Type: cd\fialab\data Press <enter>

3. At C> Type: copy *.rpt a: Press <enter>
After everything is copied - remove disc.

4. At C> Type: del *.* Press <enter>

5. Are you sure (Y/N)? Type: Y Press <enter>

6. At C> Type: cd\ Press <enter>

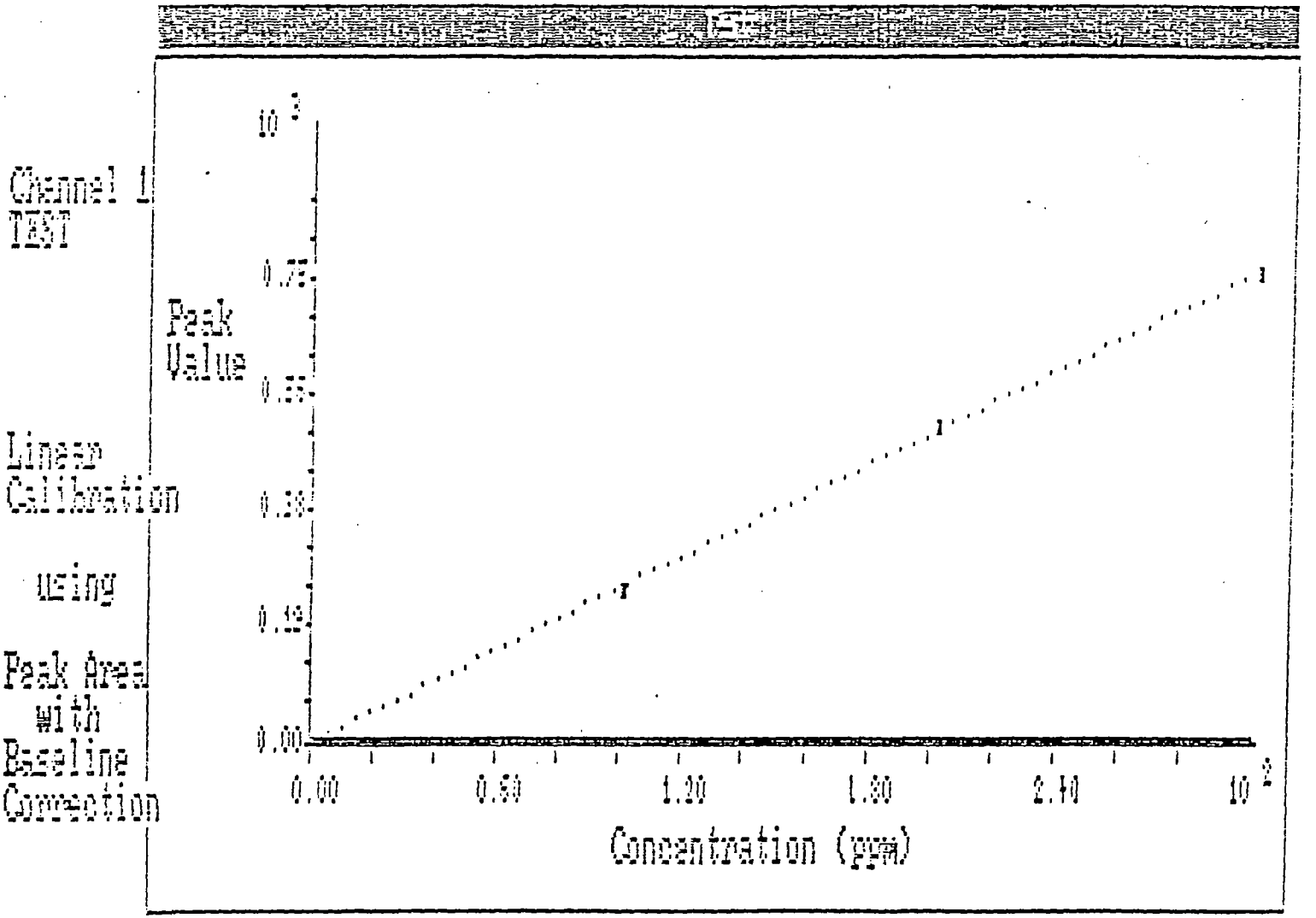
7. Turn off the red switch on the computer power strip to turn off the computer, printer and screen.

D. Quikchem Calibration

QuikCalc II uses a calibration technique called multisegment linear fitting which gives extensive flexibility to the user. It allows the calibration curve to be defined in terms of individual linear segments which can span each of several standards. Following processing of the calibration standards, a correlation coefficient is calculated for each segment with more than two standards or replicates. It provides important statistical information about each segment and gives the user a high degree of confidence in the determined sample values. See Figure 1.

Qu' 'alc II Calibration

FIGURE 1



Correlation Coefficient: 0.99925 Press the [Esc] key to continue.

RMT's RESPONSE TO COMMENTS ON SOP FOR TOC IN SOILS

RMT INC.
LABORATORIES

DATE:03/05/90

RESPONSE TO EPA QUESTIONS REGARDING RMT'S
SOP FOR TOTAL ORGANIC CARBON IN SOILS

Comment on item V:

A. The DC-80 is used in the 40 ul range only when analyzing for soils. Single point calibration is used by injecting 40 ul of 2000 ppm KHP (Potassium Acid Pthalate) standard per manufacturers' instructions.

B. 40 ul of 2000 ppm KHP is used for single point calibration per the manufacturers' instructions. Initial and continuing calibration verifications are performed with 40 ul of 2000 ppm KHP standard. A second source lab control sample (APG) is analyzed daily.
Control limits for ICV, CCV are $\pm$ 10% of true value.
Control limits for the LCS is $\pm$ 20% of true value.

Jeffrey S Gearhart
Wet Chemistry Group Leader

REVISED SOP FOR CYANIDE ANALYSIS

Effective Date 3-27-90

CYANIDE, TOTAL - DISTILLATION

Scope and Application: This method is applicable to the determination of cyanide in drinking water, surface water, groundwater, sludges, soils and industrial wastes.

Methods: Distillation, Automated Colorimetric

Reference: EPA 1983, Method 335.2

SW-846, Method 9010

Standard Methods, 16th Edition, Method 412

Detection Limit: 0.005 mg/L

Optimum Range: 0.005 - 0.40 mg/L

Sample Handling: Preserve with sodium hydroxide to pH >12 and refrigerate at 4°C. Analyze samples within 12 days.

Reagents and Apparatus:

1. Cyanide reflux distillation apparatus
2. 25 mL and 50 mL graduated cylinders
3. Vacuum pump
4. Heating mantle
5. 250 mL volumetric flasks
6. Sodium hydroxide
7. Sulfuric acid, concentrated
8. Magnesium chloride
9. Deionized water
10. Bismuth nitrate
11. Sulfamic acid
12. Acetic acid, concentrated
13. Sodium thiosulfate, crystals

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. Sodium hydroxide (1.25N): Dissolve 50.0 g NaOH in D.I. water and dilute to 1 liter in a volumetric flask. Store in a plastic bottle.

2. Magnesium chloride solution: Dissolve 510.0 g $MgCl_2 \cdot 6H_2O$ in D.I. water and dilute to 1 liter. Store in a plastic bottle.

3. Stock cyanide solution (1000 mg/L): Dissolve 0.6275 g KCN and 0.5 g KOH and dilute to 250 mls with D.I. water in a volumetric flask. Prepare fresh every month. CAUTION: TOXIC! Refrigerate.

[ACS]

4. Standard cyanide solution (5 mg/L): Pipet 5 mL of stock cyanide solution into 1 L volumetric flask containing approximately 500 mL D.I. water and 2 mL of 10N NaOH as a preservative. Dilute to volume with DI water. Prepare fresh weekly. Refrigerate.

5. Bismuth nitrate solution: Dissolve 30.0 g of $Bi(NO_3)_3$ in 100 mL of D.I. water. While stirring, add 250 mL of concentrated acetic acid. Stir until dissolved. Dilute to 1 liter with D.I. water.

6. Sulfamic acid solution: Dissolve 40.0 g of sulfamic acid in D.I. water. Dilute to 1 liter.

Notes:

1. CAUTION: Use care in handling of samples with cyanide because of the toxicity. Avoid skin contact, inhalation, or ingestion. ALWAYS HAVE A RESPIRATOR IN AREA WHEN DOING THIS TEST.

   If a sample begins to bump or back up the tube, quickly increase the flow rate, and turn the heat down (or off) until bumping subsides.

   If a sample does boil over, proceed as follows:

   - Put on respirator
   - Pull inlet tube out
   - Turn heat off (For your proctection, use gloves.)
   - Put sample and heating mantle into hood
   - When sample is cool remove from mantle and heat mantle in hood on high until acid fumes have dissapated.

2. Oxidizing agents, such as chlorine, interfere by decomposing cyanides. If chlorine is believed to present, put a drop of sample on potassium iodide starch paper. If paper turns bluish, add a few crystals of sodium thiosulfate ($Na_2S_2O_3$) to the sample, mix, and retest. Continue adding sodium thiosulfate until free from chlorine. Then, add 0.1 g sodium thiosulfate in excess.

3. Sulfides interfere by forming thiocyanate at a high pH. If sulfides are believed to be present, put a drop of sample on lead acetate test paper treated with acetic acid buffer solution at ph4. Darkening of paper indicates sulfides. If sulfides are present, add 50 mL of bismuth nitrate solution after the air rate is set through the air inlet tube. Mix for 3 minutes prior to addition of H_2SO_4.

   Alternatively, $Cd(NO_3)_2 \cdot 4H_2O$, $CdCO_3$ or $PbCO_3$ can be added after the distillation, but prior to color development. Bismuth nitrate added prior to the distillation process is the preferred choice.

4. Fatty acids, high carbonates, and aldehydes can interfere. Refer to Standard Methods for troubleshooting.

[ACS]

5. High concentrations of NO_3 and NO_2 can give false positive results. If samples contain high concentrations of NO_3 and/or NO_2, add 50 mL of sulfamic acid solution after the air rate is set through the air inlet tube. Mix for 3 minutes prior to addition of H_2SO_4.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed, and deionized rinsed prior to analyses. Dichromate or acetone may also be used to clean the glassware prior to the soap and water wash.

2. Connect and set up cyanide reflux distillation apparatus as shown in Figure 2.

3. Prepare the 0.10 mg/L cyanide digestion standard as follows:

   Add 5 mL of the 5 mg/L cyanide solution to 250 mL of DI water. (Prepare in the distillation flask.)

4. Pour 250 mL of sample into cyanide distilling flask. If a solid or semi-solid sample is to be anaylzed, use a 1.0 g sample size and add 250 mL of D.I. water to the distilling flask. (Record the amount of sample used.) Add an additional 250 mL D.I. water for a total volume of 500 mL in the distillation flask. Add 3-5 boiling chips.

   To Spike: Add 5 mL of the 5 mg/L cyanide solution to the 250 mL of sample, for a final concentration of 0.10 mg/L CN.

5. Using a graduated cylinder, add 50 mL 1.25 N sodium hydroxide to the absorber tube and connect.

6. Turn on vacuum pump and adjust so that one bubble per second enters the distillation flask through the air inlet tube.

7. Slowly add 25 mL concentrated sulfuric acid through the air inlet tube. Rinse the tube with D.I. water and wait for about 2-3 minutes, until the sulfuric acid has been dispersed into the sample.

8. Using a graduated cylinder, add 20 mL magnesium chloride solution into the air inlet tube and rinse the tube with D.I. water.

9. Turn heating mantle on to 60-63% of scale. Watch vacuum rate carefully and adjust as necessary maintaining a rate of one bubble per second. As the temperature increases, bubbling increases, and the solution can be drawn from the absorption tube or blown out the air inlet tube. Reflux for one hour after the sample comes to a boil.

10. Turn off heat and continue vacuum for 15 minutes.

11. Disconnect absorber, DI rinse absorber top into absorbing solution, and shut off vacuum pump.

12. Pour solution from absorber tube into a 250 mL volumetric flask. Using D.I. water, rinse the absorption tube (3 times) and add to the volumetric flask. Dilute to mark with DI water. Mix by inverting.

13. Distillates are ready for analysis. Proceed with Lachat SOP CNAAHC for the automated colorimetric step.

Quality Control:

1. The standard curve does not need to be carried through the distillation procedure.

2. A reagent blank is to be analyzed with each set of samples. This blank is to be carried through the distillation steps as a check for contamination. Date and initial blank container.

3. A quality control calibration standard of 0.10 mg/L cyanide is to be anaylzed with each set of samples. This standard is to be carried through the entire procedure including the distillation step. Date and initial standard container.

4. A known reference standard (LCS) is to be analyzed with each set of samples. This standard is to be carried through the entire procedure including the distillation steps. This standard must be within 80-120 % of the true value and within 95% confidence limits (if available) or the samples are to be reanalyzed. See attachment 1 for preparation imstructions.

5. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are anaylzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges.

6. Aqueous and solid/semi-solid samples are separate matrices. Duplicates and spikes are required for each matrix type.

Calculation:

1. Calculate distillate concentration with Lachat QuikChem software, in the concentration mode, using the IBM XT computer. (Be sure to calculate in any distillation dilution into the final result.)

$$\text{ug/L CN} = \frac{(\text{distillate volume,mL})(\text{distillate concentration,mg/L}) \times 1000}{(\text{sample volume,mL})}$$

$$\text{mg/kg CN} = \frac{(\text{distillate volume,mL})(\text{distillate concentration,mg/L})}{(\text{sample weight,gm})}$$

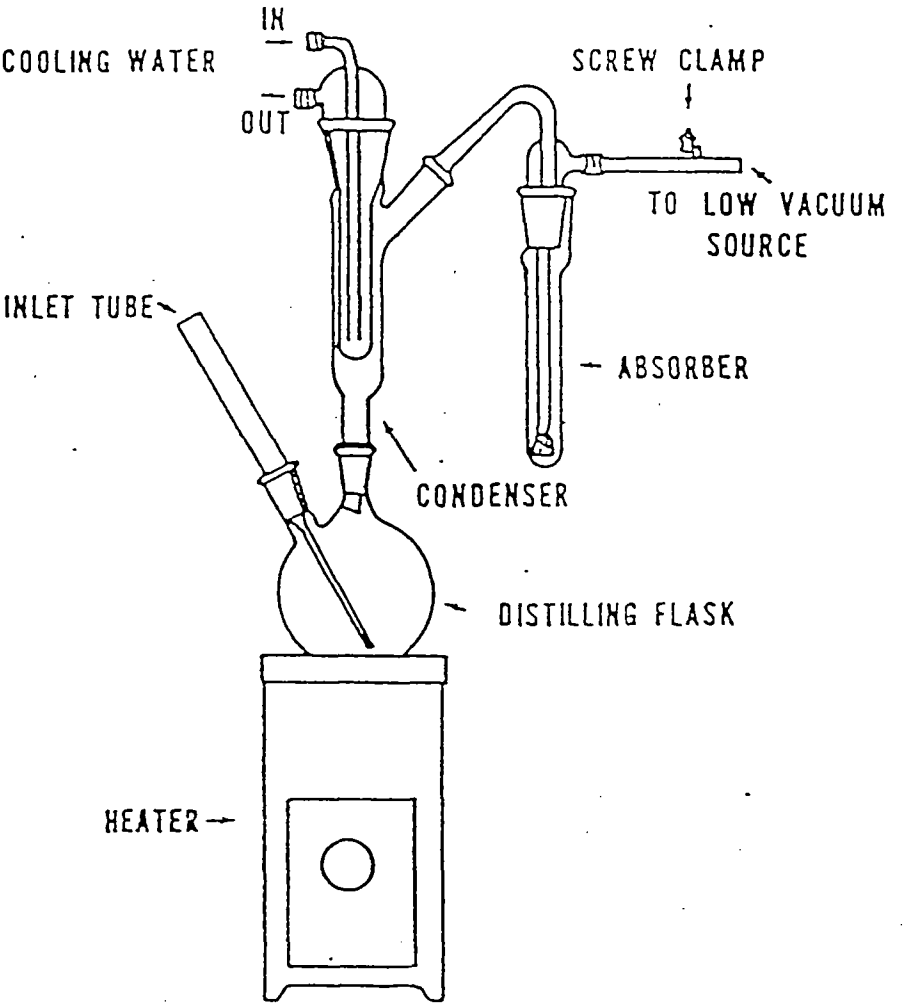


FIGURE 2

CYANIDE DISTILLATION APPARATUS

Attachment 1

Preparation of cyanide reference standards (LCS)

Environmental Protection Agency reference standard:

Add 900 ml of DI water and 2.0 mL of 10N NaOH to a 1000 mL volumetric flask. Carefully break open ampul at the mark on the neck and pipet 10.0 mL into the flask and bring to volume. Mix well. Final concentration of cyanide in lot CYN989 is 0.50 mg/L.

or

Environmental Resource Associates reference standard:

Add 900 ml of DI water and 2.0mL of 10N NaOH to a 1000 mL volumetric flask. Open concentrate vial and pipet 5.0 mL into the flask and bring to volume. Mix well. Final concentration of WasteWatR lot 9928 is 0.109 mg/L.

Final concentrations and preparation instructions vary when new lots of standard are received.

BLH/rff
[rff-ACS-cncdisc]

REVISED SOP FOR MERCURY ANALYSIS

3. Sodium chloride-hydrdoxylamine hydrochloride solution: Dissolve 120.0 g of sodium chloride and 120.0 g of hydroxylamine hydrochloride in D.I. water, dilute to 1 liter.

4. Potassium permanganate (5% solution, w/v): Dissolve 50.0 g of potassium permanganate in D.I. water, dilute to 1 liter.

5. Potassium persulfate (5% solution, w/v): Dissolve 50.0 g of potassium persulfate in D.I. water, dilute to 1 liter.

6. Intermediate mercury standard (10.0 mg/L): Transfer 1.0 mL stock mercury (1000 mg/L) solution, plus 1/2 mL nitric acid, into a 100 mL volumetric flask and dilute to the mark with D.I. water. Prepare fresh daily!

7. Working mercury standard (0.100 mg/L): Transfer 1.0 mL of the 10.0 mg/L intermediate standard, plus 1/2 mL nitric acid, into a 100 mL volumetric flask and dilute to the mark with D.I. water. Prepare fresh daily!

Notes:

1. The mercury standards are volatile and unstable. Standards must be prepared daily.

2. Because of the toxic nature of mercury vapor, precaution must be taken to avoid inhalation. Vent the mercury vapor into an exhaust hood or pass the vapor through an absorbing media.

3. A 10% solution of stannous sulfate may be substituted for stannous chloride.

4. Hydroxylamine sulfate may be used rather than hydroxylamine hydrochloride.

5. Standard additions must be used for all EP extracts and delisting petitions.

6. The calibration check standard is a 0.005 mg/L standard.

7. Interferences:

   a. Potassium permanganate is added to eliminate interferences from sulfide. Concentrations as high as 20 mg/L sulfide as sodium sulfide do not interfere.

   b. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on recovery of mercury from spiked samples.

c. Seawaters, brines, and industrial effluents, high in chlorides, will require additional potassium permanganate. during the oxidation step, chlorides are converted to free chlorine which also absorbs at the same wavelength as mercury. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. this may be accomplished by using an excess of hydroxylamine chloride reagent. In addition, the dead air space in the BOD bottle must be purged before adding the stannous sulfate.

d. Certain volatile organic materials that absorb at this wavelength may also cause an interference. A preliminary run without reagents should determine if this type of interference is present.

Instrument Conditions:

1. Wavelength: 253.6 nm. Background is required.
2. Slit Width: 0.7
3. Mode: Absorbance
4. Time = 40 seconds
5. Standards to use for curve set-up: 0.5, 1.0, 5.0, 10.0 ug/L.

Cold Vapor System Set-up:

Cell Alignment:

1. Insert quart cell in burner chamber. (Replace the burner head in the burner chamber.)
2. Align cell in light path (use 0.5 sec t, adjust to the lowest abs. reading).
3. Check drying tube and charcoal tube - replace if necessary (see attached page).
4. Insert aerator into a BOD bottle filled with 100 mLs D.I. water.
5. Turn on pump.
6. Let warm-up a few minutes.
7. Zero machine.

Procedure:

All glassware is to be washed with soap and water, rinsed with tap water, acid rinsed with 10% HNO_3, and final rinsed with D.I. water.

A. Standard Preparation

1. The standard curve is to consist of the following standards:

Standard Concentration
0.00 ug/L
0.50 ug/L
1.00 ug/L
5.00 ug/L
10.0 ug/L

2. Pipet 0, 0.5, 1.0, 5.0, and 10.0 mL aliquots of 0.10 ug/mL working stock mercury solution to 300 mL BOD bottles.

3. Add D.I. water to bring volume to 100 mL and continue with the digestion procedure.

B. Sample Preparation:

1. Transfer 100 mL, or an aliquot diluted to 100 mL, to a 300 mL BOD bottle.

   To Spike: Pipette 1.0 mL of 0.10 mg/L standard into the sample bottle. Proceed as written.

C. Digestion:

1. Add 5 mL conc. sulfuric acid and 2.5 mL conc. nitric acid to each bottle. Mix by swirling.

2. Add 15 mL potassium permanganate solution to each bottle, mix by swirling. Allow to stand for at least 15 minutes. If the bottle does not remain purple in color, additional potassium permanganate is required.

3. Add 8 mL of potassium persulfate solution to each bottle and heat for 2 hours in a water bath maintained at 95°C. Check the bottles periodically throughout the 2 hours to insure the samples remain purple. Add potassium permanganate if needed.

4. Cool to room temperature.

D. Sample Analysis:

1. Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce excess permanganate. If necessary, additional amounts of sodium chloride hydroxylamine hydrochloride may be required to discharge the purple color. Swirl.

2. Add 5 mL of stannous chloride solution and immediately insert the aerator, making sure that the stopper provides a good seal.

3. Press the read button.

4. Record the absorbance value on the bench sheet.

5. Remove the aerator, rinse aerator, and place it in the D.I. blank bottle.

6. Repeat for additional samples.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, tubing, lamp alignment, pump, etc.)

2. A quality control calibration standard of 0.005 mg/L and a blank are to be analyzed initially, and after every 10 samples. These standards are to be carried through the digestion procedure. If less than 10 samples are analyzed, a calibration standard and a blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within acceptable ranges or the samples run after the last acceptable calibration standard are to be reanalyzed. Record the calibration standard in the quality control book. The confidence limits are noted in the quality control book.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges or the data must be flagged appropriately.

Calculation:

1. Calculate using linear regression.

   Calculate the spike recovery as follows:

$$\% \text{ Recovery} = \frac{(\text{ug/L spiked sample}) - (\text{ug/L sample})}{1.0 \text{ ug/L}} \times 100$$